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PATENT ABSTRACTS OF JAPAN

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(21)Application number : 2000-217660 (71) NATIONAL INSTITUTE FOR MATERIALS SCIENCE
 (22)Date of filing : 18.07.2000 (72)Inventor : SAWADA TSUTOMU SUZUKI YOSHIHISA

(54) METHOD FOR FORMING COLLOIDAL CRYSTAL AND GELATINIZED COLLOIDAL CRYSTAL, METHOD FOR MANUFACTURING COLLOIDAL CRYSTAL ELEMENT, GELATINIZED COLLOIDAL CRYSTAL ELEMENT AND COLLOIDAL CRYSTALLINE SHEET BASED THEREON, COLLOIDAL CRYSTAL ELEMENT, GELATINIZED COLLOIDAL CRYSTAL ELEMENT AND COLLOIDAL CRYSTALLINE SHEET MANUFACTURED THEREBY

(57)Abstract:

PROBLEM TO BE SOLVED: To enable a colloidal crystal having structure excellent in three-dimensional uniformity to be formed.

SOLUTION: Colloidal crystals having structure excellent in three-dimensional uniformity are formed by giving shearing flow to a monodisperse particle solution in which particle density by volume percentage is between 5 and 70% and which is in colloidal crystalline state, and successively making the monodisperse particle solution flow in a uniaxial direction 3 in parallel to a substrate surface in a space held between two smooth substrate surfaces 11a, 11b facing each other in parallel and then making the solution stand still.



JAPANESE

[JP,2002-028471,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL
FIELD PRIOR ART EFFECT OF THE INVENTION
TECHNICAL PROBLEM MEANS EXAMPLE
DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

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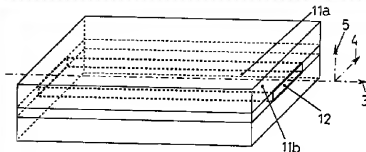
CLAIMS

[Claim(s)]

[Claim 1]Are, while particle concentration by a volume fraction is 5 to 70%, and a shear flow is given to a monodisperse particle solution which has taken a colloid crystal state, A formation method of a colloid crystal continuing at this, making it stand it still after making a monodisperse particle solution flow to 1 shaft orientations in parallel with a substrates face in space inserted into two smooth substrates faces which face in parallel, and making a colloid crystal which has the organization excellent in three-dimensional homogeneity form.

[Claim 2]A formation method of the colloid crystal according to claim 1 which sets an interval of a substrates face to 0.01-0.5 mm.

[Claim 3]A formation method of the colloid crystal according to claim 1 or 2 on which a pressure is put in parallel with a substrates face and which holds a monodisperse particle solution in a colloid crystal state, and makes it flow from the end in a straight flat tube which has a smooth substrates face which appears as two parallel sides in sectional shape.

Drawing selection **Representative draw**

[Translation done.]

[Claim 4]A formation method of the colloid crystal according to claim 3 which provides a crookedness inflow way crooked to an end or both ends of a flat tube, and makes a monodisperse particle solution flow in a flat tube through this crookedness inflow way.

[Claim 5]A formation method of the colloid crystal according to claim 1 or 2 to which puts a monodisperse particle solution in a colloid crystal state in space formed between parallel plates of two sheets which face, and relative motion of the plate of two sheets is carried out so that it may shift to linear shape momentarily to an opposite direction mutually.

[Claim 6]A formation method of a gelling colloid crystal making this gel after formation of a colloid crystal which added a gelling agent in a monodisperse particle solution, and was excellent in three-dimensional homogeneity in a formation method of the colloid crystal according to claim 1 to 5.

[Claim 7]In a formation method of the colloid crystal according to claim 1 to 5, a flat tube or a flat tube, and a crookedness inflow way are formed from transparent construction material, A manufacturing method of a colloid crystal element closing and element-izing an open end of a flat tube or a crookedness inflow way after formation of a colloid crystal excellent in three-dimensional homogeneity.

[Claim 8]A manufacturing method of a gelling colloid crystal element making this gel after formation of a colloid crystal which added a gelling agent in a monodisperse particle solution, and was excellent in three-dimensional homogeneity in a manufacturing method of the colloid crystal element according to claim 7.

[Claim 9]A manufacturing method of a colloid crystal sheet removing a flat tube and taking out a sheet shaped gelling colloid crystal after formation of a gelling colloid crystal in a formation method of the gelling colloid crystal according to claim 6.

[Claim 10]A manufacturing method of a colloid crystal sheet removing a flat tube after element-izing and taking out a sheet shaped gelling colloid crystal in a manufacturing method of the gelling colloid crystal element according to claim 8.

[Claim 11]A colloid crystal element producing by a manufacturing method of the colloid crystal element according to claim 7.

[Claim 12]A gelling colloid crystal element producing by a manufacturing method of the gelling colloid crystal element according to claim 8.

[Claim 13]A colloid crystal sheet producing by a manufacturing method of the colloid crystal sheet according to claim 9 or 10.

[Translation done.]

JAPANESE

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DETAILED DESCRIPTION

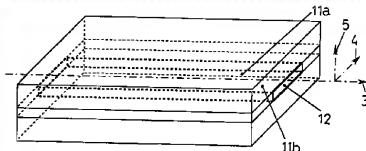
[Detailed Description of the Invention]

[0001]

[Field of the Invention]A manufacturing method of the colloid crystal element based on the formation method of a colloid crystal and a gelling colloid crystal, and this in the invention of this application, a gelling colloid crystal element, and a colloid crystal sheet. It is related with the colloid crystal element produced, a gelling colloid crystal element, and a colloid crystal sheet. A formation method of the colloid crystal in which the invention of this application can form in more detail the colloid crystal which has the organization excellent in three-dimensional homogeneity, and a gelling colloid crystal. It is related with the manufacturing method of the colloid crystal element based on this, a gelling colloid crystal element, and a colloid crystal sheet, the colloid crystal element produced and a gelling colloid crystal element, and a colloid crystal sheet.

[0002]

[Description of the Prior Art]When the particles which particle diameter is good and gathered make particle concentration in a solution high enough in the colloidal

Drawing selection **Representative draw**

[Translation done.]

solution (monodisperse particle solution) distributed into the fluid, making a periodic regular arrangement structure is known maintaining a dispersion state without particles making a fixed combination. Such particles the monodisperse particle solution which has taken the regular arrangement state, Although it is not a solid, the particles which are component units are called the colloid crystal from making three-dimensional grids structure by the analogy with the usual crystal (Tokyo Kagaku Dojin edited "colloid science I" by the Chemical Society of Japan, pp.119-123). If it stirs in the monodisperse particle solution in this colloid crystal state and a shear flow is generated, the regular arrangement structure of particles is destroyed and arrangement of particles can be changed into a disorderly state. This is called fusion of the colloid crystal. On the other hand, if it settles, particles will make an again periodic regular arrangement structure. This is called the recrystallization of the colloid crystal.

[0003]Such a colloid crystal carries out Bragg diffraction of such lights, when the grating constant is comparable as the wavelength of the ultraviolet radiation in visible light or the wavelength area of the circumference of it, or infrared light. The mirror which reflects the light filter which does not penetrate the light of specific wavelength, and a specific light using this phenomenon, Application of the colloid crystal is considered to the new optical functional material furthermore called a photonic crystal (J. D. Joannopoulos et al., Nature, Vol.386(1997)pp.143-149).

[0004]For example, the plate of two sheets made with transparent construction material as a light filter using a colloid crystal is made to face in parallel, and the element which has the structure which inserted the colloid crystal into sandwich shape in space in the meantime is known (U. S. Pat. No. 4,627,689). The colloid crystal which added the gelling agent which consists of a polymers monomer, a cross linking agent, a polymerization initiator, etc. into carrier fluid, It inserts into two substrates, after holding laminated, it is made to gel and is made a sheet shaped, and even if removing a substrate and obtaining a colloid crystal sheet is known, it is (European patent public presentation No. 0482394).

[0005]

[Problem(s) to be Solved by the Invention]In the element of the structure which inserted the former colloid crystal into sandwich shape, The monodisperse particle solution of the dissolved supercooling is put in a monodisperse particle solution as a nucleation accelerator of crystallization [**** / holding calmly to the space between parallel plates, and making it crystallize gradually spontaneously] of ion-exchange resin of a granule, is crystallized gradually, and

the colloid crystal is made to generate. However, according to such a method, although specific crystal orientation carries out orientation of the organization of the colloid crystal to crystallize to the normal line direction of a monotonous substrates face, in the direction parallel to a substrates face, it is hard to carry out orientation of it. For this reason, if several crystal grains in which oriented directions differ in process of crystallization grow simultaneously, the organization of a colloid crystal will become uneven. It is difficult to obtain a uniform organization in the colloid crystal which the generated number of a crystal grain increases, so that particle concentration is high, therefore has high particle concentration by the above-mentioned method. And since it is impossible for the very thick monodisperse particle solution with the volume fraction concentration of greater than 10% of particles to maintain a supercooling state itself, the above crystallizing methods are not employable. A colloid crystal organization tends to be disturbed by disturbance, such as vibration and a temperature change, so that particle concentration is low, and it has a crystallized state easily destroyed by the impurity elution from a container. Therefore, if it carries out from this viewpoint, to make particle concentration high is desired.

[0006]Like the above-mentioned element, the colloid crystal organization formed on the other hand although it irradiates with ultraviolet rays and gelling is made to start in the latter colloid crystal sheet does not do orientation in parallel, although the orientation of crystal orientation happens to the normal line direction of a substrates face, but the uniform crystalline structure is a stake for obtaining in three dimensions.

[0007]In three dimensions, it uses and depends for the anisotropy a uniform colloid crystal organization not only raises the performance of a light filter etc., but, and application is expected as an advanced optical functional material etc.

[0008]The invention of this application is made in view of the situation as above, and an object of the invention is to enable formation of the colloid crystal which has the organization excellent in three-dimensional homogeneity.

[0009]

[Means for Solving the Problem]An invention of this application is, while particle concentration by a volume fraction is 5 to 70% as what solves the above-mentioned technical problem, A shear flow is given to a monodisperse particle solution which has taken a colloid crystal state, After continuing at this and making a monodisperse particle solution flow to 1 shaft orientations in parallel with a substrates face in space inserted into two smooth substrates

faces which face in parallel. It is made to stand it still and a formation method (claim 1) of a colloid crystal making a colloid crystal which has the organization excellent in three-dimensional homogeneity form is provided.

[0010]About an invention concerning this claim 1, an invention of this application, In a straight flat tube which has a smooth substrates face which appears as two parallel sides in that an interval of a substrates face shall be 0.01-0.5 mm (claim 2), and sectional shape, A monodisperse particle solution in a colloid crystal state is held, and a pressure is put in parallel with a substrates face, and is made to flow from the end (claim 3), A crookedness inflow way crooked to an end or both ends of a flat tube is provided, and a monodisperse particle solution is made to flow in a flat tube through this crookedness inflow way (claim 4), A monodisperse particle solution in a colloid crystal state is put in space formed between parallel plates of two sheets which face, and it provides carrying out relative motion of the plate of two sheets so that it may shift to linear shape momentarily to an opposite direction mutually (claim 5) as a respectively desirable mode.

[0011]An invention of this application adds a gelling agent in a monodisperse particle solution in a formation method of the above colloid crystal, A formation method of a gelling colloid crystal making this gel after formation of a colloid crystal excellent in three-dimensional homogeneity (claim 6), A flat tube or a flat tube, and a crookedness inflow way are formed from transparent construction material, and a manufacturing method (claim 7) of a colloid crystal element closing and element-izing an open end of a flat tube or a crookedness inflow way after formation of a colloid crystal excellent in three-dimensional homogeneity is provided.

[0012]About an invention concerning these claims 6 and 7, an invention of this application, After formation of a colloid crystal which added a gelling agent in a monodisperse particle solution, and was excellent in three-dimensional homogeneity, A flat tube is removed after a manufacturing method (claim 8) of a gelling colloid crystal element making this gel, and formation of a gelling colloid crystal, and a manufacturing method (claim 9) of a colloid crystal sheet taking out a sheet shaped gelling colloid crystal is provided. About an invention concerning claim 8, further, an invention of this application removes a flat tube after element-izing, and provides a manufacturing method (claim 10) of a colloid crystal sheet taking out a sheet shaped gelling colloid crystal.

[0013]And a colloid crystal element, wherein an invention of this application is produced by a manufacturing method of the above-mentioned colloid crystal element (claim 11), A colloid crystal sheet (claim 13) producing by a

manufacturing method of a gelling colloid crystal element (claim 12) producing by a manufacturing method of the above-mentioned gelling colloid crystal element and the above-mentioned colloid crystal sheet is also provided.

[0014] Hereafter, it explains in more detail about an invention of this application, an example being shown.

[0015]

[Embodiment of the Invention] particles as shown in drawing 1 about the structure of the colloid crystal which the monodisperse particle solution of high particle concentration makes -- two-dimensional -- roppo, although the structure which the roppo dense layer (1) with which it was filled up densely laminates is known, When a shear flow of the strength more than fixed was given to the monodisperse particle solution in such a colloid crystal state, the tendency which carries out orientation so that the dense direction (2) of a roppo dense layer (1) or a direction (2a) (2b) equivalent to this may become parallel to a flow direction was checked. It was also checked that two substrates faces which sandwich a monodisperse particle solution and which face in parallel act so that the normal line direction and the normal line direction of the above-mentioned roppo dense layer (1) may be in agreement. Then, after making the colloid crystal made [the flow direction] to carry out orientation by shear flow flow in parallel with 1 shaft orientations in the space inserted into two smooth substrates faces which face in parallel, When it was made to stand it still, the colloid crystal was everywhere equal to two directions (3), i.e., a flow direction, and the normal line direction of the substrates face, and arranged, and it was checked that the organization excellent in three-dimensional homogeneity is obtained. The invention of this application is made based on the above technical knowledge, and completely differs in effect with the conventional common general technical knowledge. The colloid crystal was considered that it is required to eliminate disturbance, such as vibration, as much as possible, and to make it crystallize using a monodisperse particle solution with comparatively thin particle concentration until now, in order to obtain a uniform organization in big size.

[0016] When particle concentration is low, a colloid crystal dissolves easily by flow, and after a flow stop, it does not recrystallize immediately and cannot have influence of a flow on the orientation at the time of recrystallization.

Therefore, in the invention of this application, the minimum of particle concentration may be 5% in a volume fraction. Since viscous resistance will become high too much on the other hand if particle concentration becomes not much high too much, a flow becomes difficult. So, by the invention of this application, the maximum of particle concentration is

specified as 70% in a volume fraction.

[0017]Unless the flow directions of a monodisperse particle solution are 1 shaft orientations, the direction of a crystal grain will change with places and a uniform organization is not obtained. So, in the invention of this application, it is considered as 1 shaft orientations parallel to a substrates face.

[0018]When it becomes not much large too much, the above-mentioned operation stops attaining to even the inside of a colloid crystal, and if the interval of the smooth substrates face which faces becomes not much small too much, it will become difficult to function as a colloid crystal. For this reason, in the invention of this application, the interval of the substrates face makes 0.01-0.5 mm the desirable range. On the other hand, there are no restrictions in particular in the length and width of a substrates face. For this reason, the colloid crystal which was excellent in three-dimensional homogeneity covering the large area is obtained.

[0019]According to the invention of this application, also when a gelling agent is included in a monodisperse particle solution, the organization which was completely excellent in three-dimensional homogeneity by the same method is obtained. By making it gel next, the gelling colloid crystal which has the organization excellent in three-dimensional homogeneity is obtained.

[0020]When inventing this application, three kinds are illustrated concretely [the following].

[0021]In the straight flat tube (12) which has a smooth substrates face (11a) (11b) which appears as two parallel sides as the 1st in sectional shape as shown in drawing 2 and drawing 3, The monodisperse particle solution in a colloid crystal state is held, and after putting a pressure in parallel with a substrates face (11a) (11b) and making it flow from the end, making it stand it still is illustrated. A flat tube (12) provides the space inserted into two smooth substrates faces (11a) (11b) which face in parallel. If a pressure is put on the monodisperse particle solution held in the flat tube (12) from the end of a flat tube (12), a monodisperse particle solution will flow to 1 shaft orientations, i.e., the tube axial direction of a flat tube (12), parallel to a substrates face (11a) (11b) (flow direction (3)). At this time, shearing stress occurs according to the principle of hydrodynamics in the whole colloid crystal of the flat tube (12) inner whole region. The normal line direction of the roppo dense layer (1) of the colloid crystal shown in the normal line direction (5) and drawing 1 of the substrates face (11a) (11b) is in agreement, The dense direction (2) of a roppo dense layer (1), and a flow direction (3) and (the tube axial direction of flat tube (12)) will be in agreement, and the colloid crystal which has the organization excellent in three-dimensional

homogeneity will be formed in a flat tube (12).

[0022]In this case, while being able to suppress the flow distance of a monodisperse particle solution short by making into a pulse form momentary pressure the pressure put on a monodisperse particle solution, the momentary rate of flow becomes large, a shear flow momentarily strong against a monodisperse particle solution arises, and orientation of a colloid crystal is made easy.

[0023]As construction material of the cell which forms a flat tube (12), if element-ization is taken into consideration, glass will be begun and transparent plastics, such as polycarbonate, polystyrene, and an acrylic, a fused quartz, etc. will be illustrated preferably. As long as the particles of a monodisperse particle solution can exist stably in carrier fluid, the construction material is not asked. For example, organic high polymer particles, such as polystyrene and polymethylmethacrylate, are begun, and the oxide particle of various metal, such as metal particles, such as a silica particle and also gold, and silver, and titanium, and iron, etc. are illustrated. As carrier fluid, organic solvents including water, such as various alcohol and benzene, are illustrated.

[0024]By using adhesives, a board, a sheet plug, etc. and closing the both ends of a flat tube (12) after formation of a colloid crystal, it is element-ized and the whole cell serves as a colloid crystal element. Since a monodisperse particle solution has high particle concentration and its viscosity is high as above-mentioned, the tolerance over vibration of the colloid crystal element produced by doing in this way is high enough also as [this]. Similarly, a gelling agent is added in a monodisperse particle solution, and if it gels after making a colloid crystal form, it will become the gelling colloid crystal excellent in three-dimensional homogeneity, and also a gelling colloid crystal element. The gelled colloid crystal hardly receives influence in turbulence, such as vibration and a temperature change, but is very extremely stable. As a gelling agent, a proper thing can be chosen according to the construction material of the particles of a monodisperse particle solution. For example, what combined acrylamide as a polymers monomer, N-N'-methylenebis acrylamide as a cross linking agent, and the benzoin methyl ether as a polymerization initiator can be used, and gelling of a colloid crystal can be made to start by UV irradiation.

[0025]A gelling colloid crystal can be taken out as a sheet by making beforehand into decomposition ***** etc. the cell which constitutes the flat tube (12) used for formation of a colloid crystal in the case of a gelling colloid crystal furthermore, and removing. It becomes producible [a colloid crystal sheet].

[0026]When aiming at production of a colloid crystal sheet,

transparency is not necessarily required of the construction material of the cell which forms a flat tube (12). To the method of gelling, it can take making it polymerize over many hours, or heating etc. also except UV irradiation. Therefore, to aim at production of a colloid crystal sheet, element-izing is necessarily unnecessary. On the other hand, to make it gel by UV irradiation, the cell which forms a flat tube (12) needs to have transparency.

[0027]As the 2nd, as shown in drawing 4 and drawing 5, providing the crookedness inflow way (13) crooked to the above-mentioned end or both ends of the flat tube (12), making the monodisperse particle solution (14) which is in a colloid crystal state through this crookedness inflow way (13) flow in a flat tube (12), and making it stand it still is illustrated. In this case, when a monodisperse particle solution (14) passes the flection (13a) of a crookedness inflow way (13), big shearing stress occurs in a monodisperse particle solution (14) locally, and a colloid crystal carries out orientation to it in here. After a monodisperse particle solution (14) flows in a flat tube (12), the colloid crystal which has the organization which was led to the flat tube (12), flowed in parallel with a substrates face (11a) (11b), stood it still and was excellent in three-dimensional homogeneity is formed in a flat tube (12).

[0028]An injector is specifically connected with one of the crookedness inflow ways (13) via a tube. When only a few pushes in the piston of an injector rapidly, a pulse form momentary pressure is applied to a monodisperse particle solution (14) and a monodisperse particle solution (14) passes the flection (13a) of a crookedness inflow way (13), big shearing stress occurs. At first, lengthen the piston of an injector and more monodisperse particle solutions (14) on a crookedness inflow way (13) are more specifically collected. Subsequently, a piston is pushed in and operation of replacing thoroughly the monodisperse particle solution (14) which suited the flat tube (12) with the monodisperse particle solution (14) in a crookedness inflow way (13) is illustrated.

[0029]And the portion of a flat tube (12) serves as a colloid crystal element by closing the open end of a crookedness inflow way (13) using adhesives, a board, a sheet plug, etc. A gelling colloid crystal and a gelling colloid crystal element are obtained by adding a gelling agent in a monodisperse particle solution (14), and making it gel after colloid crystal formation like the 1st example. Production of a colloid crystal sheet is also attained by removal of a cell further again.

[0030]The monodisperse particle solution in a colloid crystal state is put in the space formed as the 3rd between the parallel plates of two sheets which face, and carrying out

relative motion of the plate of two sheets so that it may shift to linear shape momentarily to an opposite direction mutually is illustrated. By such monotonous relative motion of two sheets, in a monodisperse particle solution, a big shear flow occurs temporarily in one way, and a colloid crystal carries out orientation to a flow direction. Since the flow direction of the monodisperse particle solution at this time is monotonously parallel, a colloid crystal has the organization which was excellent in the three-dimensional homogeneity which carries out orientation to the same direction everywhere completely like the 1st and 2nd examples of the above.

[0031]Also in this case, a colloid crystal element, a gelling colloid crystal and a gelling colloid crystal element, and a colloid crystal sheet can be obtained like the 1st and 2nd examples of the above.

[0032]Next, the example of an invention of this application is shown.

[0033]

[Example]The cell made from a fused quartz which actually has the structure shown in [drawing 4](#) and [drawing 5](#) was produced as about 0.1 mm (t) in thickness, about 9 mm (w) in width, and about 70 mm (l) in length, and the monodisperse particle solution (14) which is in a colloid crystal state in a flat tube (12) from a crookedness inflow way (13) was poured in. . The particle concentration according [a monodisperse particle solution (14)] to a volume fraction is about 5 to 30%. It changed into the colloid crystal state by desalting enough what distributed the polystyrene particle (made by Duke Scientific) with a particle diameter of about 100-300 nm in water using positive/negative ion mixed bed type ion-exchange resin.

[0034]The colloid crystal organization obtained by the flat tube (12) became a very uniform thing which shows a uniform diffraction color except for the both ends of a flat tube (12). When the particle arrangement of the colloid crystal was observed with the confocal microscope of high magnification, it was checked that the roppo dense layer which particles build has laminated in accordance with the normal line direction (5) of the substrates face of a flat tube (12) in the direction whose one of the dense directions corresponded with the tube axial direction of a flat tube (12) in the normal line direction.

[0035]Of course, the invention of this application is not limited to the above embodiment and example. It cannot be overemphasized about details, such as construction material of the particles of a monodisperse particle solution, a kind of carrier fluid, and structure of a cell, that various modes are possible.

[0036]

[Effect of the Invention]It becomes possible to make the colloid crystal excellent in three-dimensional homogeneity form by the invention of this application as explained in detail above. The operation which the formation takes is very simple, and, moreover, serves as a short time. Since the colloid crystal formed has high particle concentration, it has high tolerance to collapse of the crystal structure by disturbance, such as vibration and a temperature change, and impurity elution, etc. Therefore, the colloid crystal element which has this colloid crystal, a gelling colloid crystal element, and a colloid crystal sheet are excellent in functionality, and also easy to manufacture.

[Translation done.]